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Dentistry — Resin-based filling materials

Art dentaire — Produits d'obturation à base de résines synthétiques

Reference number
ISO 4049 : 1988 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4049 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

This second edition cancels and replaces the first edition (ISO 4049 : 1978), of which it constitutes a technical revision (see the Introduction).

Printed in Switzerland

Introduction

This second edition of ISO 4049 takes account of the considerable volume of technical information which has accumulated since the first edition was published in 1978. Some of the tests in the first edition have been omitted and others added for the reasons given below.

This International Standard does not cover requirements for materials intended for the restoration of occlusal surfaces or those intended to prevent caries. In order to make this clear, a classification system has been introduced (see clause 3). This International Standard therefore covers class B materials, i.e. materials other than those intended for occlusal surfaces, and manufacturers are now required to classify their materials accordingly. Furthermore, in order to assist the purchaser, manufacturers are now also required (see clause 8) to describe the filler particle size range and the principal component of the resin base.

The possibility was considered that materials might be classified by filler loading or its corollary, water uptake, and solubility of the resin phase. However collaborative testing revealed considerable overlapping of these properties in "conventional" and "microfine" materials and such a classification was not adopted.

Resin-based restorative materials activated by external energy are now well established and requirements for these materials are therefore included. As the materials do not have an unlimited working time in the dental surgery, a test for sensitivity to ambient light has been included (see 7.6).

Working and setting times of chemically cured materials cannot be determined accurately because of their rapid setting and varying viscosities after mixing. The test in the first edition of this International Standard, using an oscillating rheometer, had poor sensitivity and gave results that could not be correlated with "clinical" working time. In this second edition the test has been replaced by one which is simple and widely applicable.

The flexural strength test (see 7.8) has been aligned with the test used for denture-base polymers by requiring that the specimen be immersed in water during testing. A requirement relating to modulus-dependent flexural strength has been included with the limiting value set to reveal conventional composites with poor filler/resin bonding.

Requirements have been included for materials claimed to be radio-opaque (see 4.5).

Although tests are not included in this second edition for determining non-mandatory or optional properties, such as polymerization shrinkage, it is hoped to do so in a later edition. At present more than one test may be used to determine a single such property which makes true comparisons impossible and confuses the purchaser.

The test for depth of cure of external-energy-activated materials will be reviewed and revised, if necessary, when more data become available.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that reference should be made to ISO/TR 7405 when assessing possible biological or toxicological hazards.

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Dentistry — Resin-based filling materials

1 Scope

This International Standard specifies requirements for dental resin-based restorative materials supplied in a form suitable for mechanical mixing, hand-mixing, or external energy activation, and intended for use primarily for the direct restoration of class III, IV and V cavities in the teeth, i.e. class B materials (see clause 3).

This International Standard does not cover requirements for materials intended for the restoration of occlusal surfaces, i.e. class A materials (see clause 3), or materials intended to prevent caries.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3665 : 1976, *Photography — Intra-oral dental radiographic film — Specification.*

ISO/TR 7405 : 1984, *Biological evaluation of dental materials.*

ISO 7491 : 1985, *Dental materials — Determination of colour stability of dental polymeric materials.*

ISO 8601 : 1988, *Data elements and interchange formats — Information interchange — Representation of dates and times.*

3 Classification

For the purposes of this International Standard, dental resin-based restorative materials are classified as follows :

Class A : Materials claimed by the manufacturer as suitable for the restoration of cavities involving occlusal surfaces

Class B : All other materials

Type 1 : Chemically-cured materials, i.e. those materials where setting is effected by mixing an initiator and activator

Type 2 : External-energy-activated materials, i.e. those materials where setting is effected by the application of energy, such as blue light

4 Requirements

4.1 Biocompatibility

See the Introduction for guidance on biocompatibility.

4.2 Physical and mechanical properties

4.2.1 General

If the material is supplied by the manufacturer in pre-coloured standard shades, each shade shall be capable of satisfying the

requirements specified in 4.3 appropriate to the material type. If the material is supplied for "tinting" or "blending" to the user's prescription, the material shall comply with the requirements both when used alone and when used with the maximum recommended proportion of tinter or blender [see 8.3 g)].

4.2.2 Minimum working time, type 1 materials

The working time for type 1 materials, determined in accordance with 7.4, shall be not less than 90 s.

4.2.3 Setting time, type 1 materials

The setting time for type 1 materials, determined in accordance with 7.5, shall be not more than 5 min.

4.2.4 Sensitivity to ambient light, type 2 materials

When tested in accordance with 7.6, there shall be no detectable change in the consistency of any of the three samples of type 2 materials after being exposed to the test light for 60 s.

4.2.5 Depth of cure, type 2 materials

When determined in accordance with 7.7, the depth of cure of type 2 materials shall be not less than 2 mm, and, in any event, no more than 0,5 mm below the value stated by the manufacturer.

NOTE — This test is considered to represent about twice the optimal conversion of monomer to polymer.

4.2.6 Flexural strength

The flexural strength of type 1 and type 2 materials, determined in accordance with 7.8, shall be not lower than the value of N = [(flexural modulus × 0,002 5) + 40] MPa, and, in any event, not lower than 50 MPa.

4.2.7 Water absorption and solubility, types 1 and 2 materials

When determined in accordance with 7.9, the water absorption of type 1 and type 2 materials shall not be greater than 50 μg/mm^3 and the solubility shall not be greater than 5 μg/mm^3.

4.3 Shade

When the material is assessed in accordance with 7.10 by three observers, the shade of the set material shall match closely that of the manufacturer's shade guide. If a shade guide is not supplied by the manufacturer, samples from two further batches shall be taken for comparative purposes; all three samples shall show no more than a slight change in colour.

4.4 Colour stability

When the material is assessed in accordance with 7.10, none of the three observers shall observe more than a slight change in colour.

4.5 Radio-opacity

If the manufacturer claims that the material is radio-opaque [see 7.2.3.2 b)], the radio-opacity, determined in accordance with 7.11, shall be greater than that of the same thickness of aluminium.

5 Sampling

The test sample shall consist of retail packages from the same batch containing enough material to carry out the specified tests, plus an allowance for repeat tests, if necessary.

NOTE — 50 g should be sufficient, but two further samples of different batches may be required for the shade test (see 4.3).

6 Preparation of test specimens

NOTE — For the preparation of type 2 materials, reference should be made to the manufacturer's instructions [see 8.3 e)] which will state the external energy source or sources recommended for the materials to be tested. Care should be taken to ensure that the source is in a satisfactory operating condition.

Mix or otherwise prepare the material in accordance with the manufacturer's instructions and the test conditions specified in 7.2.

7 Test methods

7.1 General reagent and apparatus

7.1.1 Water

Water prepared by

— multiple distillation, or

— distillation followed by de-ionization, or

— distillation followed by reverse osmosis.

7.1.2 Glass slides/plates

Quartz glass plates, 2 mm thick, are required for use with type 2 materials being cured by ultraviolet light only. For type 1 materials and type 2 cured by blue light, standard glass microscope slides may be used.

7.2 Test conditions

Unless specified otherwise by the manufacturer, prepare and test all specimens at (23 ± 1) °C. Control the relative humidity to ensure that it remains greater than 30 % at all times. If the material was refrigerated for storage, allow sufficient time for it to attain (23 ± 1) °C.

7.3 Inspection

Visually inspect to check that requirements specified in clause 8 have been met.

7.4 Working time, type 1 materials

7.4.1 Apparatus

Thermocouple apparatus, as shown in figure 1.

The apparatus consists of a piece of polyethylene tubing, (A), located on a block of polyamide or similar material, (B), which has a hole into which is inserted a stainless steel tube, (C), containing a stabilized thermocouple (D).

The tube (A) is 8 mm long, 4 mm in internal diameter and has a wall thickness of 1 mm. The locating part of block (B) is 4 mm in diameter and 2 mm high. When assembled the two components form a specimen well 6 mm high × 4 mm in diameter. In order to facilitate removal of the specimen after testing, the thermocouple (D) has a conical tip which protrudes 1 mm into the base of the specimen well. The tolerances on the above-mentioned dimensions are ± 0,2 mm.

The thermocouple consists of wires (0,2 ± 0,05) mm in diameter, made of a material (e.g. copper/constantan) capable of registering temperature changes in a specimen of setting material to an accuracy of 0,1 °C. The thermocouple is connected to an instrument (e.g. voltmeter or chart recorder) capable of recording the temperature to that accuracy.

7.4.2 Procedure

Prepare the test material in accordance with the manufacturer's instructions (see 8.3) and start timing from the moment mixing is begun. Maintain the mould at (23 ± 1) °C and, 30 s after the start of mixing, place the mixed material in the mould and record the temperature, t_1, of the material. Maintain the apparatus at (23 ± 1) °C and continuously record the temperature of the material until the peak temperature is passed.

NOTE — A typical recording trace is shown in figure 2. As soon as the material is inserted into the mould, the temperature falls slightly until it becomes steady at t_0 and then starts to increase. The point at which the temperature begins to increase denotes the start of the setting reaction and, therefore, the end of the working time. This point should be determined by drawing a proof line at t_0 ± 0,01 °C and recording T_w at the point of intersection with the trace. The results are extremely temperature-dependent and slight variations within the permitted temperature range will cause variations of several seconds.

Record the time, T_w, from the start of mixing until the temperature starts to increase.

Carry out five determinations.

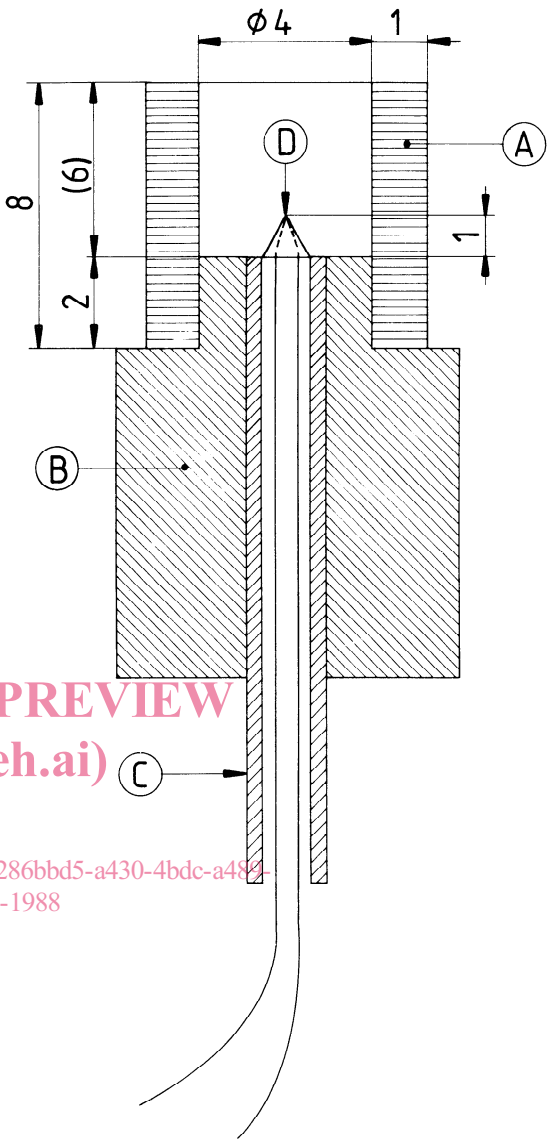
7.4.3 Interpretation of results

If at least four of the times obtained are longer than 90 s, the material is deemed to have complied with the requirement of 4.2.2.

If three or more of the times are shorter than 90 s, the material is deemed to have failed.

If only three times are longer than 90 s, repeat the whole test. If three or fewer times are longer than 90 s on the second occasion, the material is deemed to have failed the whole test.

Dimensions in millimetres

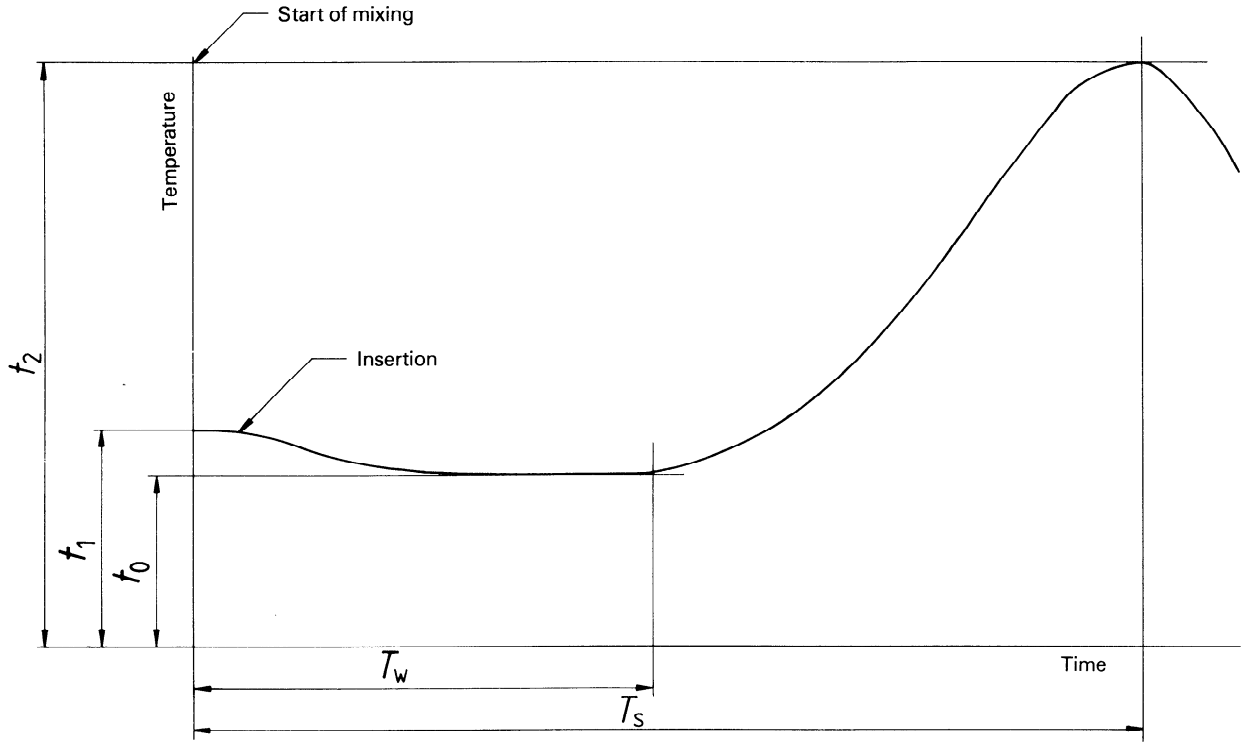


Key (see also 7.4.1)

(A) Polyethylene tubing
(B) Polyamide block
(C) Stainless steel tube
(D) Thermocouple-cone of solder

NOTE — Dimensional tolerances shall be ± 0,2 mm.

Figure 1 — Apparatus for determination of working and setting times



NOTE — The typical recording trace illustrated shows the temperature at the time of insertion, t_1, the slight temperature drop immediately after insertion, t_0, and the initial time of temperature increase, T_w, which denotes the start of the setting reaction and, therefore, the end of the working time. At (37 ± 1) °C, the peak temperature t_2 is noted to measure T_s, the setting time.

Figure 2 — Typical recording trace showing temperature changes with time for determination of working and setting times

7.5 Setting time, type 1 materials

7.5.1 Apparatus

Thermocouple apparatus, as specified in 7.4.1.

7.5.2 Procedure

Repeat the procedure specified in 7.4.2, but maintain the apparatus at (37 ± 1) °C.

Measure the time from the start of mixing until the maximum temperature is reached. Record this time, T_s, as the setting time (see figure 2).

7.5.3 Interpretation of results

If at least four of the times obtained are shorter than 5 min, the material is deemed to have complied with the requirement of 4.2.3.

If three or more of the times are longer than 5 min, the material is deemed to have failed.

If only three of the times are shorter than 5 min, repeat the whole test. If one or more times are longer than 5 min on the second occasion, the material is deemed to have failed the whole test.

7.6 Sensitivity to ambient light, type 2 materials

7.6.1 Apparatus

7.6.1.1 Xenon lamp or **radiation source of equivalent performance,** with colour conversion and ultraviolet filters inserted.

The colour conversion filter shall be 3 mm thick hardened glass and shall have an internal transmittance which matches within ± 10 % that shown in figure 3.[1)]

1) A suitable filter which corresponds to the internal transmittance shown in figure 3 and which is available commercially is the FG 15, hardened, rough-polished, 3 mm thick (supplied by Schott Glaswerke, Postbox 2480, D-6500, Mainz 1, Germany, F.R.). This information is given for the convenience of the users of this International Standard and does not constitute an endorsement of this product by ISO.

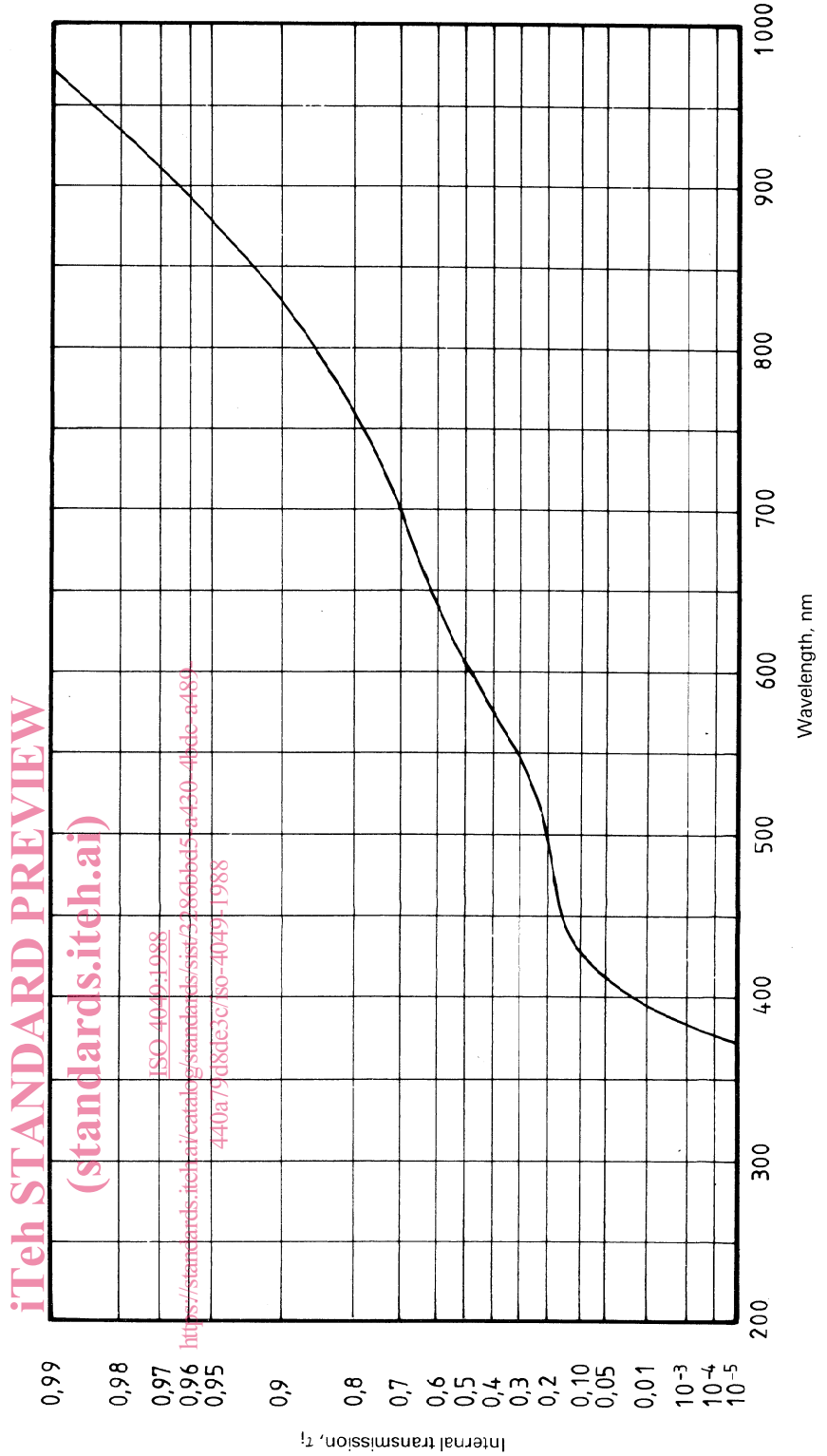


Figure 3 — Internal transmittance for colour conversion filter (see 7.6.1.1)